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HIGH TEMPERATURE DEFORMATION AND FRACTURE RESISTANCE OF SINGLE CRYSTALS OF NiAl AND NiAl-BASED INTERMETALLIC ALLOYS

Submitted to :

Department of the Air Force
Directorate of Aerospace and Materials Science
Air Force Office of Scientific Research
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Program Manager

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Table of Contents

I. Objective

The main objective of our research has been to develop a fundamental understanding of the high temperature deformation, fracture, and fatigue crack growth resistance of single crystals of NiAl and NiAl-based intermetallic alloys. Such understanding will be required for the development and implementation of constitutive laws that, in turn, will be needed to design with these materials. These alloys have shown promise as near-term alternatives to superalloys in the high temperature regime: 900-1100°C. In addition, compared to other candidate intermetallic alloys, they possess superior oxidation resistance. However, their development and use as gas turbine materials has been inhibited by limitations in both strength at high temperatures and fracture resistance at low temperatures. The research which we have conducted addresses these limitations and promises to provide a better understanding of the mechanisms that control these properties, as well as to offer possible strategies for improving these materials.

II. Approach

The overall approach we have taken in our work on intermetallics involves high resolution mechanical property studies of well-characterized and well-oriented single crystals of NiAl containing various alloying elements (Ti, Fe, Zr) in the form of both solid solutions and two phase alloys. We have studied the effects of these particular alloying elements because they are known to improve both the high temperature deformation resistance (Ti, Zr) and the room temperature fracture toughness (Fe) of NiAl. Our focus on single crystals has been based on the fact that polycrystalline materials are known to be weaker than single crystals at high temperatures. Also, these materials are highly anisotropic, both elastically and plastically, with the consequence that fundamental crystallographic mechanisms could be obscured by studying polycrystalline materials. We have conducted deformation, fracture and fatigue experiments on these materials at various temperatures and used optical and both scanning and transmission electron microscopy, as well as x-ray diffraction, to support the mechanical properties studies. We have also conducted modeling studies to understand and interpret the results of our experimental program.

Our research was characterized by close collaborations with Dr. Ram Darolia of General Electric Aircraft Engines, who graciously provided most of the single crystals we studied,

and by Drs. Anita Garg and Ronald Noebe of NASA LeRC, who provided some materials for our study and who also investigated some of our deformed crystals using TEM.

Our work on high temperature deformation was focused mainly on the basic dislocation mechanisms of creep in both single phase and two-phase Ni-Al-Ti alloys. Some of these alloys have high temperature strengths that are competitive with superalloys. Understanding the basic mechanisms that lead to these high creep strengths should provide a better basis for the design and utilization of new NiAl-based alloys for high temperature use.

Our work on fracture and fatigue crack growth resistance in NiAl-based alloys was motivated by the realization that these materials have limited ductility at low temperatures and by the knowledge that these materials will be subjected to fatigue loading environments when they are employed as high temperature structural materials. Because very little fracture toughness data were available for single crystals of these alloys and because no fatigue crack growth studies had been conducted, our work aimed to establish a reliable set of fracture and fatigue data for these single crystal materials. This work relied on the base of experience we had developed in prior work on fracture and fatigue crack growth of ceramic materials. The overall objective was to identify salient mechanisms responsible for fracture toughness and cyclic fatigue in single crystal NiAl-based alloys, to study their temperature dependence, and to develop mechanistic models describing both fracture toughness and fatigue crack growth. Based on this understanding, we expect to adapt and develop improved life prediction procedures, particularly incorporating the effects of expected service temperatures on component lifetimes. The ultimate aim of the proposed study has been to utilize these results to provide a basis for the design of NiAl alloy microstructures with optimum resistance to both fracture and cyclic fatigue.

III. Research Report

A. Mechanisms of High Temperature Deformation of NiAl Single Crystals

Keith Rory Forbes

Ph.D. Dissertation, Stanford University, March 1994.

Current interest in intermetallic alloys is based upon their potential for increased high temperature strength compared to current metals used in high temperature structural applications. An understanding of the fundamental deformation mechanisms in these alloys can provide direction for developing processes to further improve high temperature mechanical properties.

The purpose of this study has been to describe the fundamental processes of deformation in the intermetallic, NiAl. Previous studies have described easy glide with $\vec{b}$=<001> dislocations; however, this glide process is inhibited in single crystals oriented along a hard, <001> orientation. Since hard oriented crystals are ductile at high temperatures, additional deformation mechanisms must be activated. However, these additional deformation mechanisms have not been identified. Thus, in this study, special emphasis was placed on characterizing deformation in hard oriented NiAl crystals and describing the operative deformation mechanisms in these crystals.

Both hard and soft oriented single crystals of NiAl were tested in tension creep and constant strain rate compression at temperatures between 850°C and 1200°C. In addition to these monotonic tests, deformation transients in stress relaxation, strain rate change and stress change experiments were characterized for these crystals. Dislocation structures in deformed crystals were studied with transmission electron microscopy (TEM).

Soft oriented crystals show no initial transients and virtually no strain hardening in tension creep tests and constant strain rate compression tests. Transients from stress change experiments reveal that deformation is limited by the mobility of dislocations. The characteristics of deformation and observation of dislocations in soft oriented crystals are consistent with the operation of easy $\vec{b}$=<001> glide processes in these crystals.

Deformation in hard oriented crystals reveals evidence for both mobility and substructure controlled deformation. Creep in hard oriented crystals is characterized by an extensive sigmoidal transient suggesting low dislocation mobility. However, strain hardening in monotonic tests and the response of crystals in transient tests reveal that deformation is also limited by a dislocation substructure formed during deformation.

The dislocation substructure in crystals deformed along the hard orientation was observed to consist mainly of $\vec{b}$=<001> dislocations in regular networks. However, changes in shape of the sample cross-section after deformation suggest that $\vec{b}$=<101> glide is occurring. Further, Laue x-ray diffraction reveals that the networks formed in the crystal are composed of only those $\vec{b}$=<001> dislocations that would result from the decomposition of gliding $\vec{b}$=<101> dislocations by the reaction,

$$\vec{b}=[101] \Rightarrow \vec{b}=[100] + \vec{b}=[001].$$

Thus, the multiplication of $\vec{b}$=<001> dislocations during deformation is proposed to occur by the decomposition of $\vec{b}$=<101> segments. A model is developed to describe the general characteristics of $\vec{b}$=<001> network formation by $\vec{b}$=<101> glide, multiplication and decomposition. No direct evidence of $\vec{b}$=<101> dislocation interactions with $\vec{b}$=<001> networks was observed with TEM. However, TEM observation of network formation is consistent with a model of $\vec{b}$=<001> multiplication by $\vec{b}$=<101> decomposition.

Although $\vec{b}$=<101> decomposition results in a stable, low energy $\vec{b}$=<001> network, comparison of dislocation line energies reveals that the decomposition reaction is not energetically favorable. The applied stress, however, provides a driving force for decomposition that can overcome this difference in line energy. Calculations of dislocation motion under an applied <001> load reveal that $\vec{b}$=<101> dislocations with near edge character are decomposed during deformation. Dislocation segments with screw character and mixed character generally remain compact and able to glide during deformation.

Differences between deformation in hard and soft oriented crystals can thus be explained by the core structures of $\vec{b}$=<001> and $\vec{b}$=<101> dislocations. The $\vec{b}$=<001> core is compact so that glide is easy. However the $\vec{b}$=<101> core is extended out of its glide plane so that dislocation glide must involve short range diffusion and is inherently sluggish. The extended $\vec{b}$=<101> core can also be decomposed by an <001> applied load, resulting in a stable $\vec{b}$=<001> network. Thus, the increased creep resistance in hard

oriented crystals is due both to the low mobility of $\vec{b}$=<101> dislocations and to the formation of a dislocation substructure by $\vec{b}$=<101> decomposition.

B. High Temperature Deformation Behavior of NiAl(Ti) Single Crystals

Paul Harout Kitabjian,

Ph.D. Dissertation, Stanford University, April 1998.

Intermetallics are candidate materials to replace Ni-based superalloys for high-temperature structural applications, in particular the turbine blades in gas turbine engines. With proper alloying additions to improve its creep strength and ductility, NiAl-based intermetallic alloys have the potential to extend the operating temperatures of turbine engines by hundreds of degrees. Ti alloying additions have been found to be powerful strengtheners of single crystal NiAl. In this study we investigate the effects of Ti alloying additions on the high-temperature deformation behavior of NiAl(Ti) single crystals.

First, we investigate the high temperature creep behavior of the solid-solution strengthened alloys: Ni-47.5Al-2.5Ti and Ni-47Al-3Ti. Single crystals were deformed in compression in the "hard" <001> and "soft" <111> orientations, at temperatures between 827°C and 1250°C. At the highest temperatures and lowest stresses, power law creep stress exponents near three are observed, suggesting that solute drag processes may be limiting deformation at these conditions. The third power stress dependence breaks down at stresses which are expected to be high enough for dislocations to break free from solute atmospheres, again supporting the solute-drag hypothesis. The creep rates for these alloys were observed to be independent of crystal orientation and were as much as three to four orders of magnitude lower than for unalloyed NiAl. The strengthening observed is much greater, also, than is typically found in ordinary metallic solid solutions.

We attempt to understand this solid-solution strengthening effect by studying the temperature dependence of the creep rate for these alloys as well as deformation transients associated with stress changes. While the steady-state creep behavior suggests solute drag processes are controlling deformation, the creep transients are not consistent with classical solute-drag creep.

Dislocation substructures found in creep-deformed NiAl(Ti) solid solutions had forms consistent with solute-drag controlled deformation. These structures are similar to those found in classical solute-drag controlled alloy systems. It is clear from these observations that the mobility of dislocations is greatly reduced in these alloys.

The solute drag hypothesis is also supported by observations of solute size effects. An analysis of the size of Ti atoms relative to the Al atoms which they replace led to a larger study of solute size effects in NiAl-based alloys. We have determined the atomic volumes of Ni, Al and Ni vacancies in NiAl by analyzing the variations of lattice parameter with composition. We have also attempted to determine the atomic size factors for the solid-solution strengthening elements: Ti, Hf and Zr, through lattice parameter measurements of solid-solution alloys. The atomic size factor for Ti is found to be +0.34, indicating a sufficiently large size mismatch to account for significant solid-solution strengthening in this system. We calculated a similar atomic size factor of +0.27 for Ti using lattice parameters that had been computed from first-principles. Lattice contractions and negative atomic size factors were observed for Hf and Zr-containing alloys, suggesting the presence of trapped vacancies. Annealing and slow-cooling experiments minimized these lattice contractions and supported the trapped vacancy hypothesis. Using the approximation that the NiAl(Hf,Zr) lattice parameters vary linearly with composition out to the Heusler phase boundary, we determined size factors of Hf and Zr to be +0.57 and +0.65, respectively.

We attempt to determine the effect of Ti on diffusion in NiAl. Creep is a diffusive process, so changes in diffusivity have important effects on deformation behavior at high temperatures. We find that Ti reduces diffusivity in NiAl by a factor of only one half at very high temperatures; this effect alone would not provide the powerful creep strengthening observed in the solid-solution alloys.

We also examine the high-temperature deformation behavior of two-phased Ni-42.5Al-7.5Ti single crystals. Having characterized the microstructure of this alloy, we present the results of high-temperature mechanical testing, showing that this material is extremely creep resistant. The creep strengths measured compare favorably with commercial nickel-based superalloys. An examination of deformed microstructures, however, reveals that deformation in these alloys does not occur by creep. Instead, the propagation of cracks, which ultimately leads to catastrophic failure, occurs even at 1200°C.

We present models to describe the deformation behavior of NiAl(Ti) solid-solution single crystals at high temperatures that are based on the solute-drag controlled deformation. We begin with a model based on fundamental laws that govern plastic deformation. Next, we examine the classical Cottrell-Jaswon solute-drag model as it applies to this system. We modify this model using a dislocation velocity term given by James and Barnett. Finally, we present a model which incorporates both classical strengthening by dislocation interaction and structure evolution to account for the transient response of these crystals to stress changes. This model is consistent with the steady-state creep behavior and also captures the transient response of these alloys to stress changes. We conclude that strengthening in solid-solution NiAl(Ti) occurs by solute drag as well as ordinary dislocation hardening.

B. Primary Creep of $Ni_3(Al, Ta)$ Single Crystals At Low Temperatures In The Anomalous Flow Regime

Michael D. Uchic, Ph.D. Candidate

(taken from recent progress report)

Introduction

The mechanical properties of the ordered intermetallic alloy Ni_3Al have been the subject of much study over the last forty years, primarily because Ni_3Al exhibits an "anomalous" increase in flow stress with increasing temperature over a wide temperature range. Most of the work on Ni_3Al has focused on understanding the fundamental mechanism responsible for the increase in yield stress with temperature, and it is now widely accepted that this anomalous behavior can be attributed to transformations that occur within the core structure of individual dislocations. This transformation consists of the thermally-activated cross-slip of the core of screw superdislocations from {111} planes, where the superdislocation is glissile, onto {010} planes where the superdislocation is sessile. There have been many models which have been successful in explaining the details of this cross-slip event and how this process affects the yield strength of Ni_3Al, yet all of these models are based on steady-state, constant-structure conditions[1]. Chrzan and Mills have noted that "an important aspect of deformation in the $L1_2$ compounds...is the *absence* of steady-state behavior under both constant strain-rate and creep conditions," and it has been shown that these models do not accurately describe the time-dependence of plastic flow in Ni_3Al [1].

In an effort to examine the time-dependence of dislocation behavior in Ni_3Al, Chrzan and Mills developed a computer simulation which modeled the dynamic motion of a single dislocation loop under a constant applied stress (i.e. creep conditions) [1]. Their work showed that the aforementioned cross-slip pinning process effectively changed the motion of near-screw oriented superdislocations in Ni_3Al, where these dislocations moved forward by the lateral motion of mixed segments along the length of the dislocation line (these mixed segments are termed superkinks). Whereas most studies have focused solely on how the cross-slip event controls deformation in the anomalous flow regime, Chrzan and Mills showed that deformation is also controlled by the population and distribution of superkinks on a superdislocation. From their simulations, Chrzan and Mills were able to develop relations which predicted the form of the primary creep transient, and they suggested that fitting their relations to the experimentally obtained primary creep curve would allow for the extraction of *quantitative* information about dislocation motion in Ni_3Al.

Our study of dislocation dynamics in Ni_3Al has focused on providing experimental evidence to explore the predictions of the Chrzan and Mills simulations. Our primary goals have been to 1) measure the primary creep transients of Ni_3Al at different temperatures and stresses in the anomalous flow regime, and 2) correlate the shape of these experimental creep curves to the theoretical form predicted by the Chrzan-Mills model.

Experimental Procedure

The material used in this study was a Ni - 24.0 Al - 1.0 Ta alloy, where the anomalous flow regime for this composition ranges from 30 to 500°C [2]. In order to activate only one octahedral slip system, a <123> orientation was chosen for the tensile axis of the single crystal test specimen. A high-resolution tension creep apparatus (described in previous AFOSR reports) has been built to perform these creep tests at temperatures from 20 to 400°C. TEM analysis has been performed on samples after testing, where the TEM foils are prepared so that the primary glide plane of the test samples are contained within the TEM foil.

In addition to creep tests, conventional constant displacement-rate tests have been performed on the same samples. This allows for direct comparison of the mechanical behavior of Ni_3Al under different loading conditions (creep versus constant strain-rate), as

well as allowing for comparison of mechanical properties of our alloy with the results from many other research groups.

Experimental Results

Creep Test Results

We have performed creep tests on $Ni_3(Al, Ta)$ at temperatures of 20 and 100°C, where these temperatures are at the beginning of the temperature range where this alloy exhibits the anomalous flow behavior. For all stresses near and above the yield stress, the plastic response of Ni_3Al consists of a large, rapid instantaneous strain followed by a small creep strain. This creep behavior is typical of most metals which are tested both at low temperatures and at stresses above the yield stress [3]. The value of the creep rate during the rapid initial straining is on the order of 10^{-2} per second, which quickly decreases, and as shown in Figure 1, the creep rate has reached a value of $5\ 10^{-10}$ per second after 24 hours into the test. The monotonic decrease of the creep rate during the test can be expressed by the relation

$$\dot{\varepsilon} \propto t^n \qquad (1)$$

where n has been calculated to be less than -1 for all stresses and temperatures. Because the sample eventually stops deforming under creep conditions, we were able to run many creep tests on a single sample by simply incrementing the applied creep load after the sample reached a terminal strain value. Previous deformation did not appear to affect the creep behavior of Ni_3Al, as the creep curves from virgin samples and from samples that had prior deformation history showed similar behavior at the same stress.

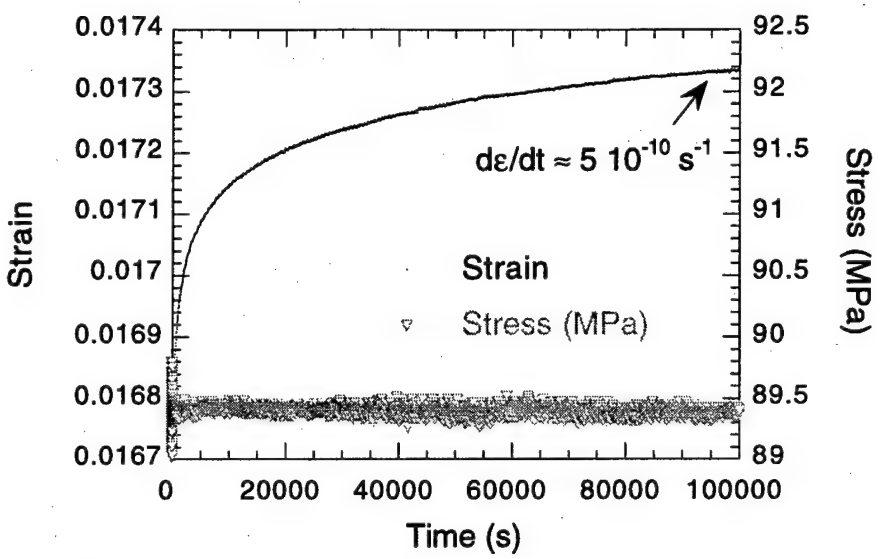

Figure 1. Creep Test At 20°C, Applied Stress Is 89.4 MPa

Of course, we would like to know how the creep behavior of Ni_3Al changed with changes of either the applied stress or temperature. The effect of increasing the applied stress increased both the amount of overall strain as well as the amount of time-dependent creep strain for both test temperatures. The effect of increasing temperature (for the same applied stress) decreased both the amount of overall strain and the amount of creep strain--the creep behavior of Ni_3Al was anomalous in nature. As shown in Equation 1, the creep curves for all tests exhibited creep rates that declined according to a power law form. For the room temperature experiments, the power law exponent, n, did not show any strong correlation with changes in the applied stress. However, for the 100°C tests, a definite correlation between the applied stress and the power law exponent was found, namely, that the power law exponent grew more negative as the applied stress was increased (see Figure 2). This indicates that these samples crept to exhaustion faster at the highest stresses, and we are currently working to understand this creep behavior in terms of the dislocation model that Chrzan and Mills have put forth.

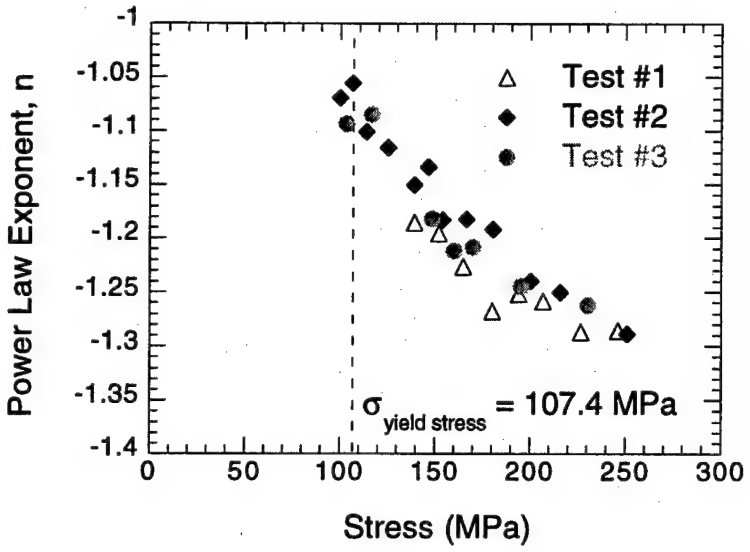

Figure 2. Power Law Exponent (n) As A Function of Stress, 100°C Tests

In addition, we are also working on fitting the experimental creep curves to the theoretical relations suggested by Chrzan and Mills in order to extract quantitative information about dislocation dynamics in Ni_3Al, and have had some initial success with this technique.

Delayed Yield Behavior

As mentioned in the previous section, many creep curves were obtained from one sample by incrementing the applied stress after a sample had been crept to exhaustion (where the strain rate has reached a value of less than 10^{-8} s^{-1}). For the 20°C tests, the sample did not respond immediately to the increase in stress when the magnitude of the stress increment was below 6 MPa, and there was a measurable lag time before the sample plastically flowed to the expected strain value. This is shown in Figure 3, where the maximum value of the strain rate occurred 42 seconds after the stress was increased by 4.3 MPa. The response time of the delayed plastic flow phenomena appeared to be most sensitive to the increment in the creep stress, where longer delay times were observed for smaller stress increments. This delayed plastic response was rarely observed for the 100°C tests. For these tests, the sample started to plastically flow at a slow rate (strain rates of less than 10^{-4} per second) immediately after a small stress increment, and the creep rate monotonically decreased for the course of the test. Although we cannot yet explain the origin of the delay time phenomena, we believe that this behavior is linked to the thermally-activated remobilization of previously immobile dislocation segments, and we are currently working on a model to explain this unexpected behavior.

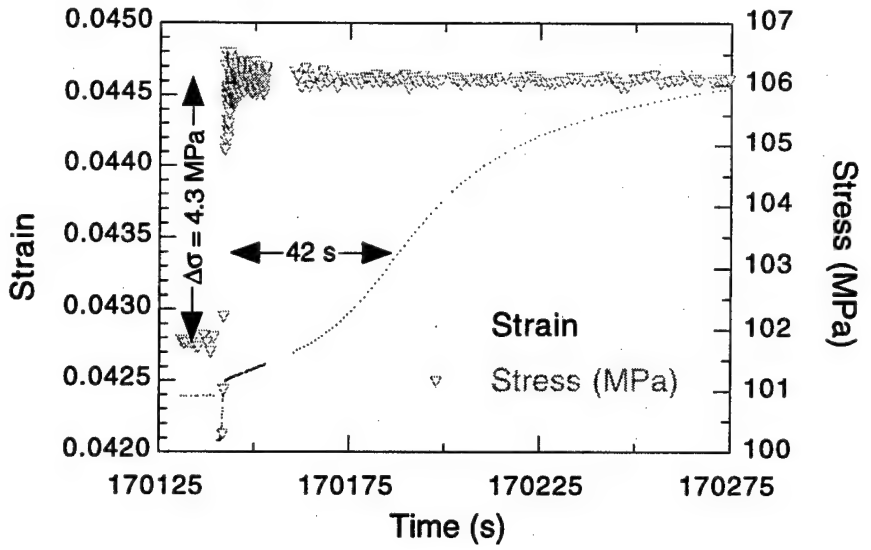

Figure 3. Delayed Plastic Response To Increment In Applied Stress, 20°C Creep Test

TEM Results

The TEM micrographs of creep tests at 20 and 100°C show microstructures dominated by a homogeneous distribution of long, near-screw oriented dislocations connected by mixed dislocation segments. These images closely resemble the dislocation microstructures obtained in Ni_3Al under constant strain rate conditions for the same test temperatures [4]. Because the post-mortem microstructures are very similar for the two different types of loading conditions (creep and constant strain-rate), we believe that our creep experiments are a valid method for exploring the same dislocation dynamics responsible for the yield strength anomaly.

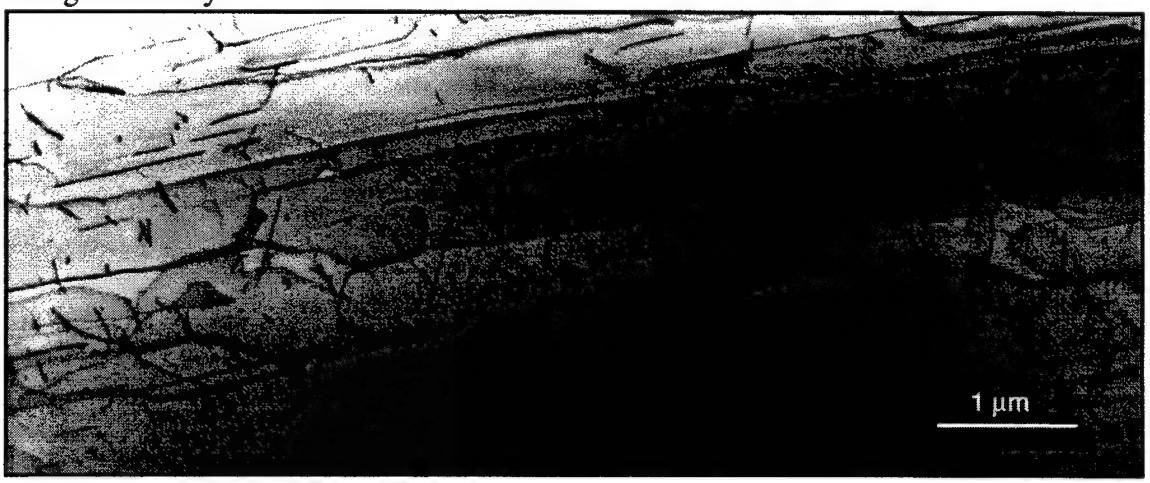

Figure 4. Bright Field TEM Image Of 20°C Creep Test, Sample Strained to 6%

Summary

Tension creep experiments have been performed on Ni_3Al at two temperatures in the anomalous flow regime. The observed creep behavior shows primary creep leading to eventual exhaustion at all stresses and temperatures. The decline of the creep rate with time has been measured as a function of temperature and the applied stress. The reinitiation of plastic flow after creep exhaustion has been shown to have a time dependence for small stress increments for the room temperature tests. TEM observations of creep tested samples show microstructures that are very similar to the microstructures observed in constant strain-rate tested samples.

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D. Fracture and Fatigue Crack-Growth Behavior of Intermetallic Single Crystals

Katharine M.Flores, Ph.D. Candidate

(taken from recent progress report)

Objectives

Our research on the fracture and fatigue crack-growth behavior of single crystal intermetallics was motivated by the realization that these materials have limited ductility at low temperatures and that they will be exposed to severe fatigue loading during service. This work explores the fundamental micromechanisms of both fracture and cyclic fatigue crack-growth in single crystal NiAl. Particular emphasis has been placed on subcritical

crack growth under principally cyclic applied loads at temperatures as high as 1100°C. The objective has been to identify mechanisms responsible for cyclic fatigue, to study their temperature dependence, and to develop mechanistic models for describing fatigue failure. Based on this understanding, toughening strategies and improved life prediction procedures may be developed, particularly incorporating the effects of expected service temperatures and crack size on component lifetimes.

Accomplishments

1. Introduction

Single crystal intermetallics have significant potential for gas turbine applications where high temperature strength and oxidation resistance are required. With its low density, high thermal conductivity, and superior oxidation resistance, NiAl + X is a leading candidate as an alternative to conventional superalloys. Typical gas turbine applications will expose the material to cyclic and vibrational loading. However, very limited reliable fracture and subcritical crack-growth data currently exists, together with little fundamental understanding of damage and failure modes for NiAl single crystals, particularly at elevated temperatures. There is virtually no knowledge of what constitutes a fracture or fatigue-resistant microstructure or composition, and there is a real uncertainty in potential life-prediction procedures. Accordingly, this report describes progress made over the last year in developing fracture mechanics based techniques to characterize fracture and fatigue crack-growth behavior in a NiAl + Fe single crystal at elevated temperatures. We believe the fatigue crack-growth data presented in this report to be quite unique representing the first reported subcritical crack-growth data at elevated temperatures in such intermetallic single crystals.

2. Experimental Work

The limited fracture toughness data available for NiAl single crystals at both room and elevated temperatures is often compromised by the use of blunt notches rather than atomically sharp cracks, and violation of minimum thickness requirements for plane strain conditions at elevated temperatures. It has been noted that using sharp cracks instead of blunt notches lowers both the brittle to ductile transition temperature and the fracture toughness for some specimen orientations (Bergmann and Vehoff, Scripta Met, 1994). It has also been determined that various alloying additions, such as iron, can increase the room temperature ductility of NiAl (Darolia et al, Scripta Met, 1992). The material selected

for initial study, therefore, was a 50Ni-49.75Al-0.25Fe compound provided be GE. Fracture toughness and fatigue crack-growth behavior was measured following sample preparation and pre-cracking to produce an atomically sharp fatigue crack.

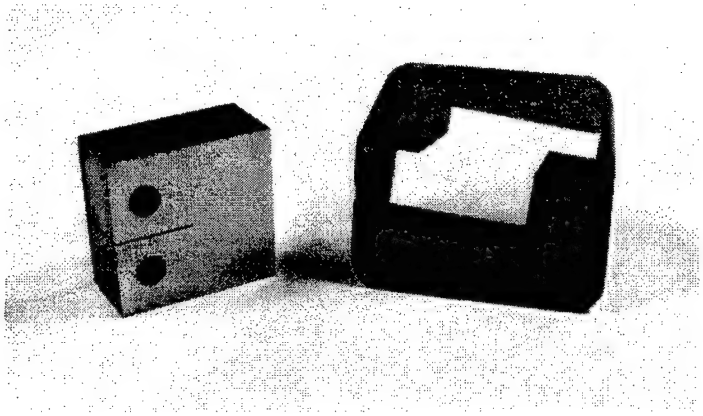

Figure 1. Compact tension geometry employed in study.

Sample Preparation and Pre-Cracking: The bi- and tri-crystalline slabs provided by GE had been homogenized at 2400°F for 50 hours. Following crystal orientation determination by Laue X-ray diffraction, compact tension samples were prepared by wire EDM (Fig. 1). The specimen dimensions were designed according to the specifications of ASTM Standard E399-90, for *Plane-Strain Fracture Toughness of Metallic Materials.* Initial studies have focused on cracks propagating on {110} cleavage planes in <001> directions. The plastic zone size was estimated for this crack orientation by calculating the resolved shear stress on the <100>{001}, <100>{011}, and <111>{112} slip systems, which are expected to be active at room temperature, and the <110>{011} slip system, which replaces <111>{112} above approximately 330°C. The largest radius of these expected plastic zones was used to determine the required specimen thickness to ensure plane strain conditions at a given temperature. Room temperature specimens were 3 mm thick. Specimens tested at 800°C were slightly more than 6 mm thick. Fracture toughness and fatigue-crack propagation rates (da/dN) were determined in general accordance with the current ASTM Standards E399 and E647, respectively, using a procedure specifically developed for crack growth testing in brittle materials.

Testing was performed on a high resolution, computer controlled electro-servo-hydraulic test system. Crack lengths for the room temperature tests were continually monitored using 20 μm thick NiCr bonded crack gauges in addition to compliance and optical techniques to

a resolution of ~2μm. The specimens were loaded at 25 Hz during the room temperature fatigue tests. For the elevated temperature tests, crack lengths were monitored using compliance techniques, and the specimens were loaded at 10 Hz to ensure that the extensometry could adequately follow the applied waveform. Room temperature pre-cracking proved to be the most challenging portion of the test and was achieved using a combination of tensile fatigue cycles and compressive overloads. For both pre-cracking and subsequent fatigue testing at room and elevated temperatures, the specimens were loaded with a load ratio of 0.1 under automated load-shedding schemes. The fatigue behavior was monitored under decreasing ΔK conditions.

3. Results

The results of cyclic fatigue crack-growth tests at $R = 0.1$ are plotted in Figure 2 in terms of the growth rate per cycle, da/dN, as a function of the stress intensity range, ΔK. Both room temperature results reported previously and those at 800°C are shown for comparison. To the best of our knowledge, this represents the only reported cyclic fatigue crack propagation behavior for NiAl single crystals at either room or elevated temperatures. Room temperature crack growths ranged over two orders of magnitude from 10^{-8} to 10^{-10} m/cycle with fatigue threshold value ΔK_{TH} of approximately 2.3 MPa$\sqrt{m}$. The elevated temperature results cover only one decade. Gross deformation of the sample forced the end of the test before a threshold could be reached.

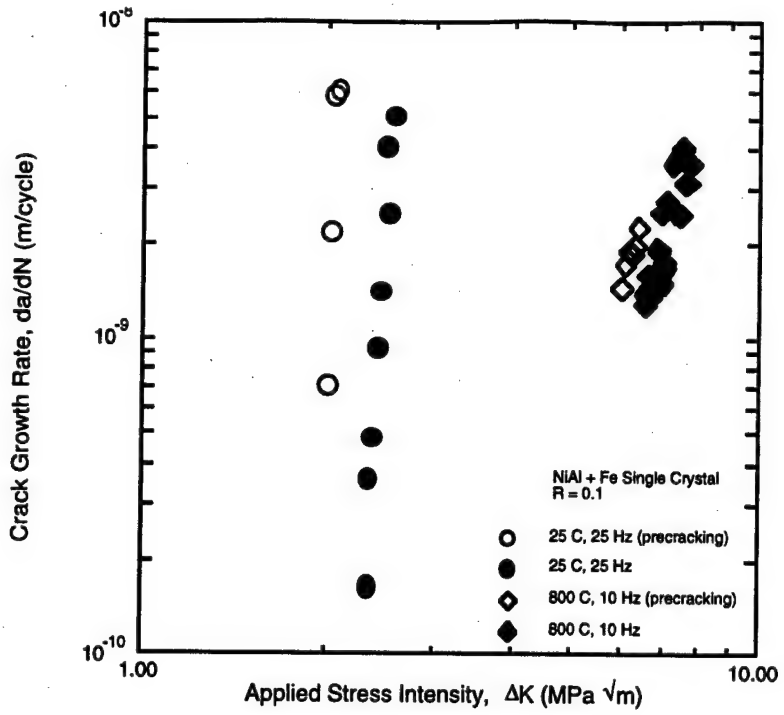

Figure 2. Fatigue crack growth data for NiAl+ Fe single crystal.

Note that the slope of the room temperature data is much greater than that at elevated temperatures. We may fit this data with a power law,

$$\frac{da}{dN} = C\Delta K^m$$

The exponent, m, for the room temperature data is quite high, on the order of 34, which is typical of very brittle materials. At elevated temperatures, the single crystal intermetallic begins to behave more like a normal metal, with an exponent on the order of 6. This change in behavior is a result of the increased ease of dislocation motion at higher temperatures.

The fractography of the room temperature specimens has been discussed previously. While room temperature tests seemed to indicate that fatigue crack growth was occurring on the {112} or {111} crystallographic plane, with slip occurring on the easy <100>{001} system, no such change in crack plane was evident in the specimens tested at 800°C. An SEM micrograph of the transition from a room temperature fatigue region to an 800°C

fatigue region is shown in Figure 3. Note the marked increase in roughness in the elevated temperature region.

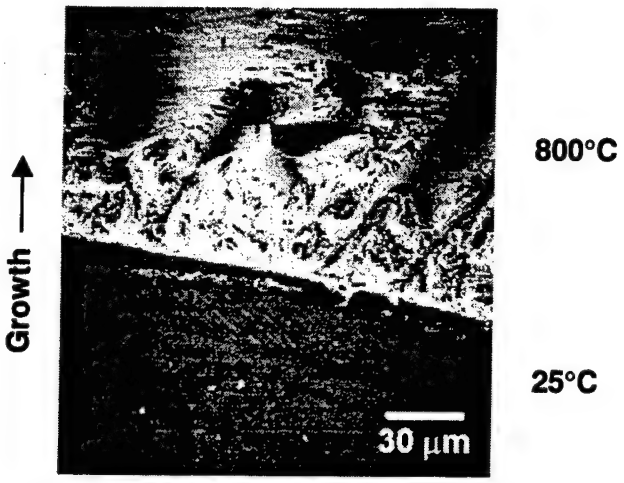

Figure 3. Transition from room temperature (25°C) to elevated temperature (800°C) fatigue.

While the crack did not appear to change planes, slip on the <100>{001} system is again evident. The increase in fatigue surface roughness with temperature is a result of increasing ease of dislocation motion at higher temperatures. The parallel, relatively smooth planes oriented at about a 45° angle with the main crack plane in the 800°C growth region appear to be {001} planes, consistent with the activation of the easy <100>{001} slip system. The exposed part of the slip planes cover a much greater area in the high temperature fatigue region than in the room temperature fatigue region, indicating that dislocations were able to travel greater distances at the higher temperature, giving rise to the rougher surface.

IV. Publications and Oral Presentations

A. Publications resulting from AFOSR Grant Nos. AF-F49620-95-1-0163, 92-J-0009-P00001 AND 89-0201

1. P.H. Kitabjian, A. Garg, R.D. Noebe and W.D. Nix, "High-Temperature Deformation Behavior of NiAl(Ti) Solid-Solution Single Crystals," (accepted for publication in Materials Transactions)

2. P.H. Kitabjian and W.D. Nix, "Atomic Size Effects in NiAl-Based Solid Solutions," Acta Materialia,46, 701-710 (1998).

3. K.J. Hemker and W.D. Nix, "Mechanisms of Dislocation Creep in Single Phase Nickel and Titanium Aluminides," Proceedings of the International Symposium: Structural Intermetallics 1997, Eds. M.V. Nathal et al., The Minerals, Metals and Materials Society, 1997, p.21-32.

4. M.D. Uchic and W.D. Nix, "Transient Creep of Ni3(Al,Ta) Single Crystals at Room Temperature," Proceedings of the Sixth International Conference on Creep and Fracture of Engineering Materials and Structures, Irvine, CA, Eds: J.C. Earthman and F. A. Mohamed, The Minerals, Metals and Materials Society, 1997, p.269-278.

5. P.H. Kitabjian, A. Garg, R. D. Noebe and W.D. Nix, "High-Temperature Creep Behavior of Single Crystals of the Solid-Solution Alloy NiAl-2.5Ti," Proceedings of the Sixth International Conference on Creep and Fracture of Engineering Materials and Structures, Irvine, CA, Eds: J.C. Earthman and F. A. Mohamed, The Minerals, Metals and Materials Society, 1997, p.667-676.

6. P.H. Kitabjian, A. Garg, R. Noebe and W.D. Nix, "The Solid-Solution Alloying Effects of Ti on the High Temperature Deformation Behavior of NiAl Single Crystals," Materials Research Symposium Proceedings, 460, 479-486 (1997).

7. M.D. Uchic and W.D. Nix, "Primary Creep of Ni3(Al,Ta) Single Crystals at Room Temperature," Materials Research Symposium Proceedings, 460, 437-442 (1997).

8. K. M. Flores and R. H. Dauskardt, "Fracture and Fatigue Crack-Growth Behavior of Single Crystal NiAl," Scripta Materialia, 36 [12] 1377-1382, 1997.

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13. K.R. Forbes, U. Glatzel, R. Darolia and W.D. Nix, "High Temperature Deformation of Single Crystals of NiAl", Materials Research Symposium Proceedings, 288, 45-57 (1993).

14. K.R. Forbes and W.D. Nix, "Transient Deformation of Single Crystal NiAl at High Temperatures", Materials Research Symposium Proceedings, 288, 385-390 (1993).

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B. Oral Presentations resulting from AFOSR Grant Nos. AF-F49620-95-1-0163, 92-J-0009-P00001 AND 89-0201

1. K.J. Hemker and W.D. Nix, "Mechanisms of Dislocation Creep in Single Phase Nickel and Titanium Aluminides," International Symposium: Structural Intermetallics 1997, Seven Springs Mountain Resort, PA, September 21-25, 1997. (invited lecture)

2. P.H. Kitabjian, A. Garg, R. D. Noebe and W.D. Nix, "High-Temperature Creep Behavior of Single Crystals of the Solid-Solution Alloy NiAl-2.5Ti," Proceedings of the Sixth International Conference on Creep and Fracture of Engineering Materials and Structures, Irvine, CA, August 15, 1997.

3. W.D. Nix, "Dislocation Channeling, A Robust Concept for Understanding Strength of Thin Films, Multilayers and Other Finely Structured Materials," Department Colloquium, Mechanical and Environmental Engineering, University of California, Santa Barbara, February 24, 1997 (invited lecture)

4. P.H. Kitabjian, R.D. Noebe, A. Garg and W.D. Nix, "The Solid Solution Alloying Effects of Ti on the High Temperature Deformation Behavior of NiAl Single Crystals," MRS Symposium: High Temperature Ordered Intermetallic Alloys, Materials Research Society, Fall Meeting, Boston, MA, December 4, 1996. (poster presentation)

5. M.D. Uchic and W.D. Nix, "Primary Creep of Ni3(Al,Ta) at Low Temperatures in the Anomalous Flow Regime," MRS Symposium: High Temperature Ordered Intermetallic Alloys, Materials Research Society, Fall Meeting, Boston, MA, December 4, 1996.

6. W.D. Nix, "High Temperature Deformation and Fracture Resistance of Single Crystals of NiAl and NiAl-based Intermetallic Alloys," AFOSR Structural Metals Workshop, Bar Harbor, Maine, August 20-22, 1996.

7. W. D. Nix, "Mechanisms of High Temperature Deformation of NiAl Single Crystals," International Symposia on Advanced Materials and Technology for

the 21st Century, The Japan Institute of Metals, Honolulu, Hawaii, December 14, 1995

8. W.D. Nix, "Mechanisms of High Temperature Deformation of NiAl Single Crystals", Department Colloquium, Materials Science and Engineering, The Ohio State University, Columbus, Ohio, May 19, 1995.

9. W.D. Nix, K.R. Forbes and K.J. Hemker, "High Temperature Creep of Intermetallic Alloys", The 7th JIM International Symposium on Aspects of High Temperature Deformation and Fracture in Crystalline Materials, The Japan Institute of Metals, Nagoya, Japan, July 25, 1993.

10. K.R. Forbes and W.D. Nix, "Mobility and Substructure Controlled Creep Deformation of [001] Oriented NiAl Crystals", The Fifth International Conference on Creep and Fracture of Engineering Materials and Structures, The Institute of Metals, Swansea, Wales, March 29, 1993.

11. K.R. Forbes, U. Glatzel, R. Darolia and W.D. Nix, "High Temperature Deformation of Single Crystals of NiAl", MRS Symposium on High Temperature Ordered Intermetallic Alloys V, Materials Research Society Fall Meeting, Boston, MA, December 1, 1992.

12. K.R. Forbes and W.D. Nix, "Transient Deformation of Single Crystals of NiAl at High Temperatures", MRS Symposium on High Temperature Ordered Intermetallic Alloys V, Materials Research Society Fall Meeting, Boston, MA, December 2, 1992. (poster presentation).

13. U. Glatzel, K.R. Forbes and W.D. Nix, "TEM Observations of Dislocation Structures in Single Crystals of NiAl after High Temperature Creep", MRS Symposium on High Temperature Ordered Intermetallic Alloys V, Materials Research Society Fall Meeting, Boston, MA, December 2, 1992. (poster presentation).

14. K.R. Forbes and W.D. Nix, "Creep in NiAl Alloys", High Temperature Materials Workshop, WI/MLLM, April 8-9, 1992.

15. U. Glatzel, K.R. Forbes and W.D. Nix, "Orientation Dependence of Intermediate and High Temperature Deformation in Single Crystal NiAl", General Abstract Session: Intemetallics, Annual Meeting of the Metallurgical Society, San Diego, CA, March 2, 1992.

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18. K.J. Hemker and W.D. Nix, "An Investigation of Creep in Ni3Al(B,Hf)", Fourth International Conference on Creep and Fracture of Engineering Materials and Structures, Swansea, Wales, April 2, 1990.

19. W.D. Nix, ""Advances in Understanding High Temperature Strengthening of Metals and Alloys", 1989 Edward deMille Campbell Memorial Lecture, Annual Meeting of ASM International, Indianapolis, Indiana, October 3, 1989.

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